

Original Article

Spectrophotometric Determination of Ni(II) Ion with 7-Bromo-2-Nitroso-1-Oxinaphthalene-3,6-Disulphocid

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Received: 20 March 2024

Revised: 16 May 2024

Accepted: 27 May 2024

Published: 29 June 2024

Abstract - In this article, the spectrophotometric detection method of complexing Ni(II) ion with 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulphocid (BNOX-S, S-3,6) (HR) with organic analytical reagent developed and studied optimal conditions: $\lambda_{max} = 640$, pH = 6,5, universal buffer, reagent - buffer - Ni(II) - distilled water, BNOXS-S with 0.05% relative to $T_{Ni^{2+}} = 10 \mu\text{g}/25\text{ml}$, 1.0 ml of S-3.6 reagent was found to be sufficient. The area of obedience to the Bouguer-Lambert-Beer law was determined to be 1.0-17.5 $\mu\text{g}/25 \text{ ml}$. Absorption spectra were studied: sensitivity, according to Sendel, was 0.0011 $\mu\text{g}/\text{cm}^2$, contrast $\Delta\lambda = 100 \text{ nm}$. The composition of the complex and the mechanism of complex formation were studied using the Nazarenko method, isomolar series method, Asmus' straight-line method, and spectrophotometric titration methods. The molar ratio of the complex is Me: R=1:2. The actual molar extinction coefficient ($\epsilon_{real} = 50000$), the formation equilibrium constant ($K_{equilibrium} = 9.71 \cdot 10^{-5}$) of the complex formed by Ni(II) with BNOXS-S, S-3,6, the stability of the complex constant using the Babko method ($\lg\beta = 18,87$), the confidence interval of the deviation from the mean value ($\Delta X = 0.104$) and the lower limit of detection ($Q_{minimum} = 0.203 \mu\text{g}/25\text{ml}$) were determined. The results of the graduated graph were mathematically processed using the method of small squares, and a linear mathematical equation was developed: $Y_i = 7,7 \cdot 10^{-3} + 1,78 \cdot 10^{-2} X_i$. The effect of foreign ions was studied. The developed method was used in an artificial mixture. In this case, the relative standard deviation (Sr) did not exceed 0.021.

Keywords - Ni(II) ion, 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulphocid, Organic analytical reagent.

1. Introduction

Today, as a result of the continuous development of the industry, including the operation of enterprises, factories, and plants, many chemical compounds and toxic metals that cause environmental pollution are contaminating rainwater and wastewater as ecotoxicants[1]. These ecotoxicants include copper, cadmium, nickel, and other heavy metals. It is important to identify metals from sources contaminated with these metals and to reduce their amount or even completely clean them [2]. The optimal conditions for spectrophotometric determination of Ni(II) ions using D-penicillamine have been investigated[3].

This study outlines simultaneous spectrophotometric techniques for quantifying Ni²⁺ ions using 1-(2-pyridylazo)2-naphthol (PAN) in micellar media. The ligand and its metal complexes, namely Ni²⁺-PAN, were rendered water-soluble with the neutral surfactant Triton X-100, eliminating the need for organic solvent extraction[4]. The spectrophotometric technique was employed to investigate the interaction between Ni(II) and Cu(II) ions with three different ligands: ethyl 4-(4-hydroxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine

-5-carboxylate (Ligand 1), 4-(1 H-benzimidazol-2-yl)phenol (Ligand 2), and 2-(3-phenylamino-4,5-dihydro-1,2-oxazol-5-yl)phenol (Ligand 3). These interactions were studied under conditions of 0.01 M ionic strength and 28°C in a 70% dioxane-water mixture [5]. The compound 2-(2-Thiazolylazo)-p-cresol (TAC) undergoes a reaction with Ni(II) ions within the pH range of 6–10, resulting in the formation of a blue complex in a 1:2 ratio. The stability constant of this complex is determined to be 5.0 ± 0.3 [7,8].

A method was developed for the extraction and concentration of nickel in the form of its dimethylglyoxime complex using triethylamine. The nickel-enriched triethylamine extract was then separated and evaporated. The resulting residue was dissolved in chloroform (50 μL), and the absorbance at 380 nm was measured. A spectrophotometric procedure for the determination of nickel was established, with a limit of detection of 0.020 $\mu\text{g}/\text{mL}$ ($n = 10$; $P = 0.95$) [9,10].

The aim of this study is to determine the optimal conditions for the reaction of complex formation of Nickel (II)



with 7-bromo-2-nitroso-1-oxynaphthalene-3, 6-disulfoacid and to develop a method of rapid, sensitive, and selective spectrophotometric determination based on it.

2. Experimental Part

2.1. Reagents and Solutions

Standard solutions of Nickel(II) salts with a titer of $T_{Ni^{2+}} = 1000$ mg/ml were prepared using accurately weighed portions of analytical grade metallic mineral salts. "Clean" and "chemically clean" brand reagents were used in the experiment. An accurately weighed portion of nickel(II) salt was placed in a 1000 mL volumetric flask and dissolved in distilled water. Small-concentration solutions were prepared by serial dilution of the prepared standard solution. Reagent solutions are prepared by dissolving 0.0500 g of reagent with distilled water in a 100 ml volumetric flask.

2.2. Methods

Spectrophotometric studies of colored solutions were carried out on an EMC-31PC-UV spectrophotometer. The pH of the solutions was controlled using a pH meter (pH-150M OAO, Gomelsky zavod izmirtelnyx priborov) with an accuracy of ± 0.05 pH units. One of the main conditions of the complex formation reaction is the environment of the solution. Therefore, buffer solutions with different pH values were used to obtain reproducible results. For this, 5 ml of a universal buffer solution with a pH value of 2.46 to 11.2, 0.05% 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulfoacid (BNOKS-S, S-3,6) is added to a 25 ml measuring flask reagent solution, 1.0 ml of 40 $\mu\text{g/ml}$ Ni(II) solution was added and diluted by adding distilled water up to the mark of the flask.

The optical density of the complex compound solution was measured in an EMC-31PC-UV spectrophotometer at a wavelength of $\lambda_{\text{max}}=640$ nm in cuvettes with an absorption thickness of $\ell=1.0$ cm. The obtained results are presented in Figure 1.

According to the results presented in Figure 1, the highest optical density of the complex compound was observed in the range of pH 5.8-7.0. The highest optical density was chosen at pH=6.5. During the study, the effect of buffer solutions of different compositions with the same pH=6.5 on the light absorption of the color complex was studied. The results are presented in Figure 2.

According to the results presented in Figure 3, the color complex showed the highest optical density under the influence of a universal buffer solution [11,12].

During the research, the time dependence of the formation of a colored complex was studied. According to the obtained results, the fact that the optical density of the formed colored complex did not change for 4 hours indicates that there is enough time for the analysis. When the order of pouring was studied, reagent-buffer-Ni(II)-distilled water produced the highest optical density in the order. To form a complete

complex of the metal ion, the amount of reagent is taken in excess; for this purpose, the dependence on the amount of reagent was studied (Figure 3). According to the research results, it was found that 1.0 ml of BNOKS-S, S-3.6 reagent with 0.05% $T_{Ni^{2+}}=10$ $\mu\text{g}/25\text{ml}$ is enough.

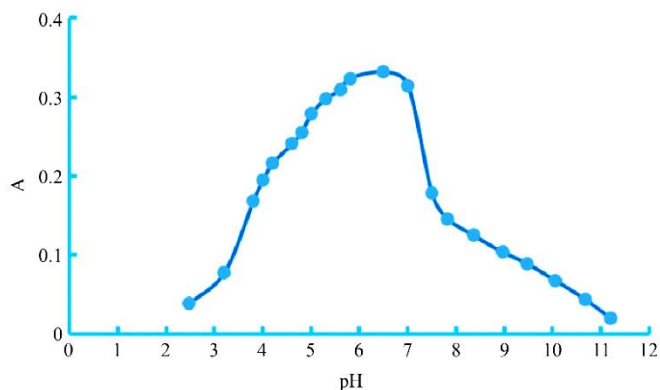


Fig. 1 Graph of dependence of optical density on solution environment. ($\lambda_{\text{max}}=640$ nm, $\ell=1,0$ cm, $n=5$)

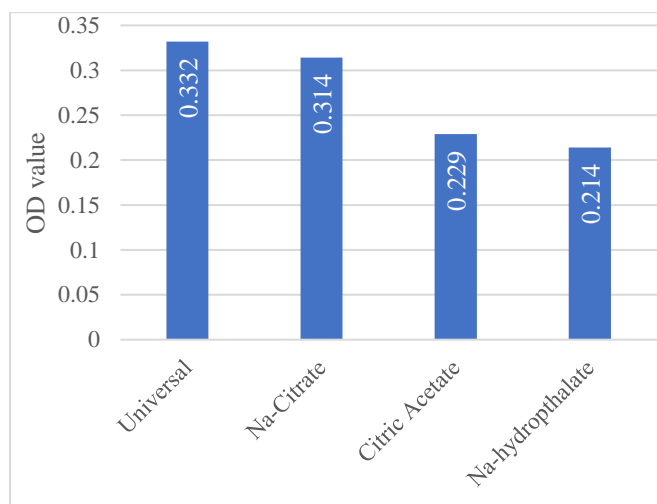


Fig. 2 Graph of dependence of optical density on buffer solution content ($\lambda_{\text{max}}=640$ nm, $\ell=1,0$ cm, pH=6,5, $n=5$)

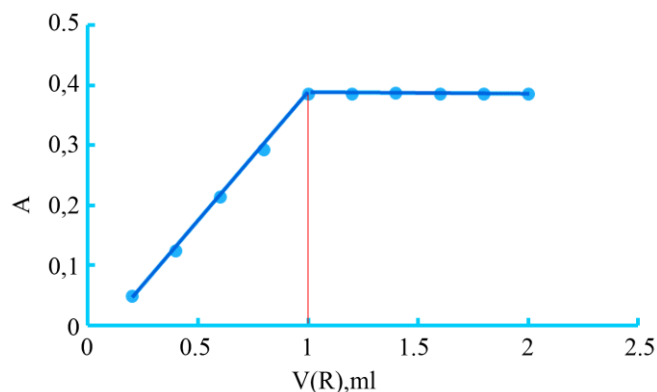


Fig. 3 The graph of the dependence of the optical density of the complex compound on the amount of added reagent. ($\lambda_{\text{max}}=640$ nm, $\ell=1,0$ cm, pH=6,5, $n=5$).

3. Results and Discussion

3.1. Bouguer-Lambert-Beer Law

1.0 ml of 0.05% BNOKS-S, S-3,6 aqueous solution, 5.0 ml of universal buffer solution pH=6.5, variable amount of 5 µg/ ml of Ni(II) standard solution was diluted to the mark of the flask by adding distilled water. By mixing the solutions, their optical densities were measured in comparison to the reference solution. The obtained results are presented in Figure 4.

According to the obtained results, compliance with the Bouguer-Lambert-Beer law was observed in the range of 1.0-17.5 µg/25ml. At higher concentrations, there was a deviation from the linear relationship[13].

3.2. Absorption Spectra

UV-visible spectra were obtained under optimal conditions using the BNOKS-S, S-3,6 reagent and the complex formed with Ni(II) (Table 1). According to the method's sensitivity by Sendel, the light absorption per unit area (0,001 of mkg/sm²) was calculated using the following formula:

$$S. b. s = \frac{Q \times l \times 0.001}{A \times 25} = \frac{15 \times 1 \times 0.001}{0.568 \times 25} = 0.0011 \text{ mkg/sm}^2$$

Where: S.b.s - Sensitivity according to Sendel, Q - Concentration of the absorbed metal ion, l - Path length of the cuvette, A - Optical density.

Based on the results obtained during the research, it is evident that the reaction exhibits both a considerable contrast ($\Delta\lambda=100 \text{ nm}$) and high sensitivity ($S.b.s=0.0011 \text{ µg/cm}^2$). The composition and mechanism of complex formation are determined. By utilizing the Nazarenko method, it was established that the shape of the complex involving Ni(II) is Ni^{2+} [15,16]. The stoichiometry of the complex under investigation was determined using the Isomolar series method (Figure 5), Asmus' correct linear method (Figure 6), and the spectrophotometric titration method (Figure 7). All the methods employed in the research confirmed that the molar ratio of components in the complex is Me: R=1:2.

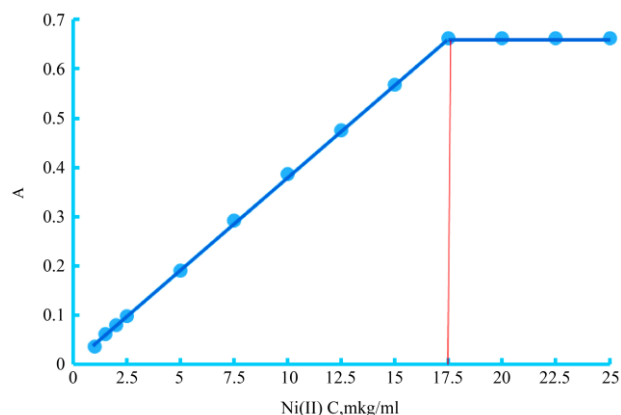


Fig. 4 Graph of dependence of the optical density on the amount of added Ni(II). ($\lambda_{\text{max}}=640 \text{ nm}$, $l=1.0 \text{ sm}$, $\text{pH}=6.5$, $n=5$)

Table 1. Spectral characterization of the BNOKS-S,S-3,6(HR) and its complex with Ni(II) (MeR) ($C_{\text{Ni}^{2+}}=15 \text{ mkg}$, $\lambda_{\text{max}}=640 \text{ nm}$, $l=1.0 \text{ cm}$, $\text{pH}=6.5$, $n=5$)

Complex color	pH	Maximum wavelength, nm		$\Delta\lambda$	$C_{\text{Ni}^{2+}}$, mkg/	$C_{\text{Ni}^{2+}}$,	\bar{A}	Sensitivity, according to Sendel, $\mu\text{g/cm}^2$
		MeR	HR		25 ml	mol/l		
Kompleks Ni(II) bilan R ($l=1.0 \text{ sm}$, $n=5$)								
Blue-green	6,5	640	540	100	15	$1,02 \times 10^{-5}$	0,568	0,0011

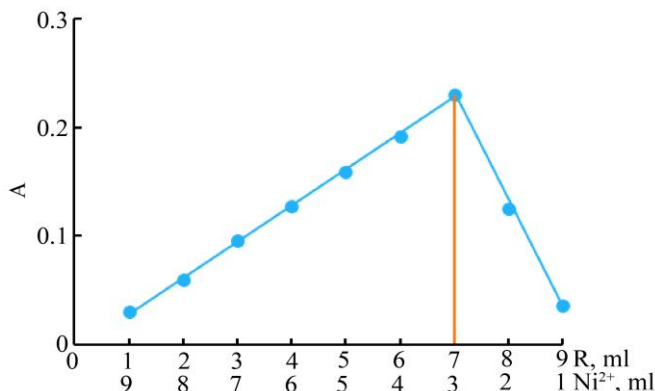


Fig. 5 Isomolar series method graph of the composition of the complex formed by Ni(II) with BNOKS-S, S-3,6. ($\lambda_{\text{max}}=640 \text{ nm}$, $l=1.0 \text{ cm}$, $\text{pH}=6.5$, $n=5$).

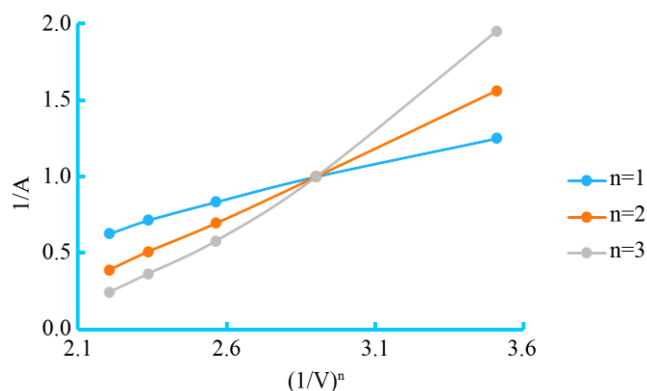


Fig. 6 Graph of determining the composition of the complex formed by Ni(II) with BNOKS-S, S-3,6 using Asmus' correct linear method. ($\lambda_{\text{max}}=640 \text{ nm}$, $l=1.0 \text{ cm}$, $\text{pH}=6.5$, $n=5$).

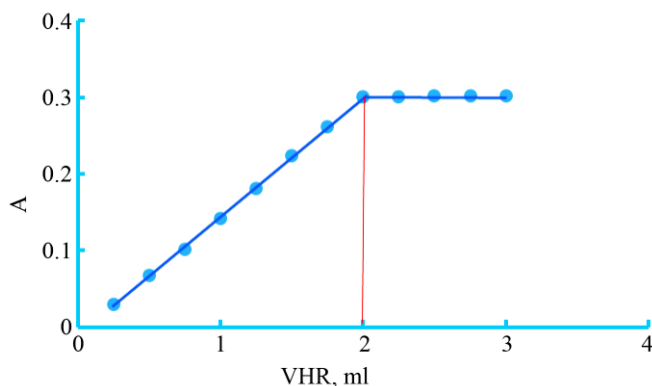


Fig. 7 Graph of the spectrophotometric titration method. ($\lambda_{max}=640$ nm, $l=1.0$ cm, $pH=6.5$, $n=5$).

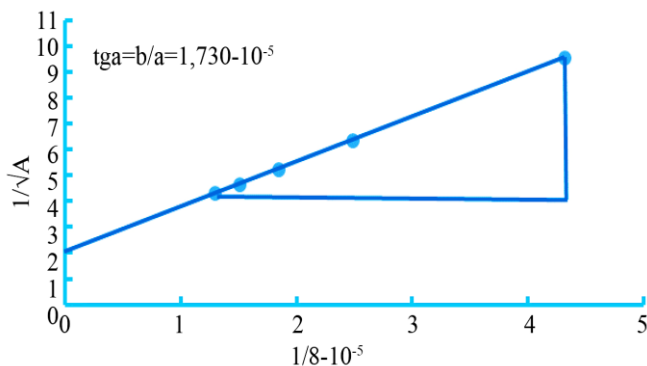


Fig. 8 Graph for determining the molar extinction coefficient. ($\lambda_{max}=640$ nm, $l=1.0$ cm, $pH=6.5$, $n=5$)

To thoroughly investigate the complex formation reaction of Nickel (II) with the BNOKS-S, S-3,6 reagent, the main characteristics of the complex, namely the true molar extinction coefficient and the formation equilibrium constant, were determined using Tolmachev's graphical method (Figure 8). Additionally, the stability constant of the complex was determined using Babko's method (Table 2).

$$\epsilon_{haq} = \frac{1}{1/\epsilon \cdot 10^n} = \frac{1}{2,0 \cdot 10^{-5}} = 50000$$

$$K_{muv} = \frac{C_H^n \cdot l^n}{n^n \cdot \epsilon_x \cdot B^{n+1}} = \frac{(3,16 \cdot 10^{-7})^2 \cdot 1^2}{2^2 \cdot 50000 \cdot (1,73 \cdot 10^{-5})^3} = 9,71 \cdot 10^{-5}$$

Where C_H - Concentration of H^+ ions, l - Cuvette thickness, ϵ_{real} - True molar extinction coefficient of the complex, n - Stoichiometric coefficient.

$$K_{unstable.(MeR)} = \frac{[H^+] \cdot (A_1 \cdot C_2 - A_2 \cdot C_1)^{q+1}}{(C_1 \cdot \sqrt[3]{A_2} - C_2 \cdot \sqrt[3]{A_1}) \cdot (A_1 \cdot \sqrt[3]{A_2} - A_2 \cdot \sqrt[3]{A_1})^q}$$

$$K_{stable} = \frac{1}{K_{unstable.(MeR)}}$$

Where $[H^+]$ - Hydrogen ion concentration, A_1, A_2, A_3 - Optical densities, C_1, C_2, C_3 - Metal ion concentrations.

Based on the calculated values of $\epsilon_{haq}=50000$, $K_{equilibrium}=9.71 \times 10^{-5}$, and $lg\beta=18.87$, it was determined that the method developed has high sensitivity, while the complex has an average stability.

The results obtained from the graph with calibration were mathematically reprocessed using the method of least squares, and a linear mathematical equation was constructed (Table 3 and Figure 9).

From the obtained results, the calibration graph (calibrated graph) equation was $Y_i=a+bX_i$ ya'ni $Y_i=7,7 \cdot 10^{-3} + 1,78 \cdot 10^{-2}X_i$. The accuracy and repeatability of the determination were calculated using the "entered-found" method.

The relative standard deviation (Sr) value did not exceed 0.03, the confidence interval relative to the average value (ΔX) was 0.104, and the lower limit of determination (Qmin) did not exceed 0.203 $\mu g/25ml$ [14, 17].

Table 2. Results of determining the stability constant of the complex using Babko's method. ($\lambda_{max}=640$ nm, $l=1.0$ cm, $pH=6.5$, $n=5$)

№	$V_{Ni^{2+}}$, ml	V_R , ml	$C_1 \cdot 10^{-6}$	A_1	$C_2 \cdot 10^{-6}$	A_2	$K_{unstable} (MeR)$	$K_{stable} (MeR) (\beta_k)$	$lgK_{stable} (MeR) (lg\beta_k)$
1	0,5	1	5,11	0,054	2,555	0,026	$1,09 \cdot 10^{-21}$	$9,16 \cdot 10^{20}$	20,96
2	1	2	10,2	0,161	5,1	0,070	$3,48 \cdot 10^{-19}$	$2,88 \cdot 10^{18}$	18,46
3	1,5	3	15,3	0,294	7,65	0,123	$1,50 \cdot 10^{-18}$	$6,67 \cdot 10^{17}$	17,82
4	2	4	20,4	0,441	10,2	0,198	$6,39 \cdot 10^{-19}$	$1,57 \cdot 10^{18}$	18,19
5	2,5	5	25,6	0,592	12,8	0,281	$1,29 \cdot 10^{-19}$	$7,75 \cdot 10^{18}$	18,89
Σ							$5,23 \cdot 10^{-19}$	$1,86 \cdot 10^{20}$	18,87

Table 3. Results of constructing and reprocessing the calibration graph ($\lambda_{max}=640$ nm, $l=1.0$ cm, $pH=6.5$, $n=5$, $P=0.95$)

№ T/r	Cu ²⁺ mkg, Xi	\bar{A} , Yi	Xi ²	Xi · Yi	Y _i _{hisob.}	Yi-Y _i _{hisob.}	(Yi-Y _i _{hisob.}) ² · 10 ⁻⁶
1	2	0,045	4	0,090	0,0433	0,00153	2,34
2	6	0,114	36	0,684	0,1145	-0,00048	0,226
3	8	0,150	64	1,197	0,1501	-0,00044	0,196
4	10	0,185	100	1,850	0,1857	-0,00068	0,462
5	12	0,222	144	2,659	0,2213	0,00033	0,109
6	14	0,254	196	3,553	0,2569	-0,00307	9,440
7	17,5	0,322	306,25	5,635	0,3192	0,00281	7,900
Σ	69,5	1,291	850,25	15,668	1,2909	-	20,7

Table 4. Results of determining Ni(II) ions from a synthetic sample ($\lambda_{max}=640$ nm, $l=1.0$ cm, $pH=6.5$, $n=5$, $P=0.95$)

Composition of the mixture, µg	Entered Ni ²⁺ , µg	A	Found Ni ²⁺ , µg X _i	S	S _r	$\bar{X} \pm \Delta X$
K ⁺ , Na ⁺ (600); Bi ³⁺ , Co ²⁺ (0,2); NO ₂ ⁻ , Cl ⁻ (60); Hg ²⁺ (0,02); Ba ²⁺ , NO ₃ ⁻ (800); CH ₃ COO ⁻ (200);	12,0	0,223	11,96	0,252	0,021	12,10 ± 0,313
		0,230	12,35			
		0,227	12,18			
		0,219	12,74			
		0,229	12,29			

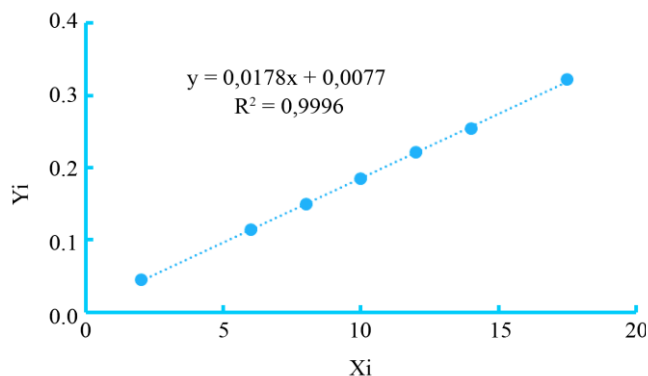


Fig. 9 Calibrated graph for the determination of Ni(II).

$$Q_{min} = \frac{5 \cdot S_a \cdot M \cdot V \cdot B \cdot 1000}{\epsilon \cdot l}$$

$$= \frac{5 \cdot 0,001386 \cdot 1 \cdot 25 \cdot 58,6934 \cdot 1000}{50000 \cdot 1}$$

$$= 0,203 \text{ mkg}/25\text{ml}$$

Where ϵ_x - true molar extinction coefficient, V - volume of the sample, B - atomic mass of the element, l - cuvette

thickness, M - number of atoms of Ni(II) present in the complex composition, Sa - standard deviation, Q_{minimum}-lower limit of determination.

In the spectrophotometric analysis of Ni(II) ions (15 µg) using the BNOKS-S, S-3,6 reagent, interfering foreign ions were investigated. It was determined that the following ions did not interfere with the detection of Ni(II) ions at the specified ratio: K⁺, Na⁺, NH₄⁺, Ba²⁺, Citrate, F⁻, Cl⁻, J⁻, Br⁻, SO₄²⁻, NO₃⁻, ClO₄⁻, PO₄³⁻, Cr₂O₇²⁻, C₂O₄²⁻, CH₃COO⁻; Pb²⁺(1:1000), Pb²⁺, Ti⁴⁺, Si₂O₃²⁻ (1:900); Al³⁺, Mn²⁺, Cr³⁺, NO₃⁻, Ni²⁺(1:7,5); Cu²⁺, Bi³⁺(1:5); Sn²⁺(1:1). The interfering ions are as follows: Co²⁺, Cd²⁺, Fe²⁺, Mg²⁺, Ca²⁺, Fe³⁺, S₂O₃²⁻, EDTA, thiomorpholine, and SCN⁻ (1:0,5). The interfering ions are neutralized using the precipitation method [6].

To verify the accuracy and repeatability of the method, the "entered-found" method was employed using a synthetic sample (Table 4). In order to neutralize interfering foreign ions in the determination of Ni(II) ions, Cl⁻ and NO₂⁻ ions were added in a ratio of 1:60. According to the obtained results, it is possible to detect Ni(II) in such naturally occurring samples. The relative standard deviation (Sr) did not exceed 0.021.

4. Conclusion

According to the results, the optimal conditions for the formation of the complex of Ni(II) ions with the BNOKS-S, S-3,6 reagent were determined. The range of applicability according to the Bouguer-Lambert-Beer law is 1.0-17.5 $\mu\text{g}/25\text{ml}$, with a sensitivity (S.b.s) of 0.0011 $\mu\text{g}/\text{cm}^2$ and a wavelength shift ($\Delta\lambda$) of 100 nm. The Isomolar series method, Asmus' correct linear method, and spectrophotometric titration method confirmed the composition of Me: R=1:2.

Using Tolmachev's graphical method, the true molar extinction coefficient ($\epsilon_{\text{haq}}=50000$) and the formation equilibrium constant ($K_{\text{miv}}=9.71 \cdot 10^{-5}$) were determined. Additionally, using Babko's method, the stability constant of the complex ($\lg\beta=18.87$) was determined. A calibrated graph (calibration curve) with a linear equation $Y_i = 7,7 \cdot 10^{-3} + 1,78 \cdot 10^{-2} X_i$ was constructed. The confidence interval relative to the average value (ΔX) was determined to be 0.104, and the lower limit of determination (Q_{min}) was found to be 0.203 $\mu\text{g}/25\text{ml}$. Interfering ion effects were investigated.

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Acknowledgment

The authors thank Termez State University and the Termez Institute of Engineering and Technology for supporting this research work.

Authors' Declaration

We hereby confirm that all the Figures and Tables in the manuscript are ours.

Ethical Clearance

The project was approved by the local ethical committee at the Termez Institute of Engineering and Technology and Termez State University.

Authors' Contribution Statement

N.Sh.S. conducted the research, T.Kh.Kh. the conception, design, and drafting, N.B.A. was responsible for the acquisition of data Sh.R.S. did the interpretation; K.Sh.A. participated in the conception, design, and drafting and all the authors took part in revision and proofreading.

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